

Investigation of moisture condition and Autoclave sensitivity on air permeability measurements for both normal concrete and high performance concrete



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HIGHLIGHTS

- We study variations of relative humidity and electrical resistance after drying and conditioning.
- We investigate influences of moisture content and distribution on results of the Autoclave air permeability.
- We examine the sensitivity of the Autoclave air test to measure permeability of HPCs.
- Drying in an oven at 50 °C and 35% RH for 14 days can eliminate the effect of moisture variation on the Autoclave air test.
- The Autoclave air test is unable to differentiate the differences between HPCs, but the BS-EN water penetration test does.

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ABSTRACT

While on site measurement of air permeability provides a useful approach for assessing the likely long term durability of concrete structures, no existing test method is capable of effectively determining the relative permeability of high performance concrete (HPC). Lack of instrument sensitivity and the influence of concrete moisture are proposed as two key reasons for this phenomenon. With limited systematic research carried out in this area to date, the aim of this study was to investigate the influence of instrument sensitivity and moisture condition on air permeability measurements for both normal concrete and HPC.

To achieve a range of moisture conditions, samples were dried initially for between one and 5 weeks and then sealed in polythene sheeting and stored in an oven at 50 °C to internally distribute moisture evenly. Moisture distribution was determined throughout using relative humidity probe and electrical resistance measurements. Concrete air permeability was subsequently measured using standardised air permeability (Autoclave) and water penetration (BS EN: 12390-8) tests to assess differences between the HPCs tested in this study.

It was found that for both normal and high performance concrete, the influence of moisture on Autoclave air permeability results could be eliminated by pre-drying (50 ± 1 °C, RH 35%) specimens for 3 weeks. While drying for 5 weeks alone was found not to result in uniform internal moisture distributions, this state was achieved by exposing specimens to a further 3 weeks of sealed pre-conditioning at 50 ± 1 °C. While the Autoclave test was not able to accurately identify relative HPC quality due to low sensitivity at associated performance levels, an effective preconditioning procedure to obtain reliable air permeability of HPC concretes was identified.

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1. Introduction

Use of high-performance concrete (HPC) is an established approach to enhancing the durability of reinforced and pre-stressed concrete structures [1,2].

However, with performance levels of HPC typically based on laboratory-based testing [1], the long term, in service performance of concrete structures is largely dependent on contributing factors

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such as construction quality. Against this background, an ability to undertake accurate in situ quality assessment of HPC is very important.

Most concrete durability problems are directly related to the ingress of various aggressive substances, such as Cl^- , SO_4^{2-} and CO_2 [3,4] and numerous techniques have been developed to measure related permeation properties in order to assess/predict structural durability [5,6]. Although water permeability tests are suitable for in situ performance assessment, air permeability tests have gradually become popular due to their simplicity, short test durations and lack of physical/chemical interaction during measurements [5,7].

While many researchers [8–10] have undertaken HPC air permeability assessments using available in situ techniques, reported results have not been conclusive. As air permeability is affected by concrete's pore structure and moisture condition, one explanation for this is the influence of moisture. As there is no standard preconditioning regime for field methods, most in situ experiments are carried out under site-specific moisture conditions. Without appropriate preconditioning methods, the influence of moisture is likely to be a significant cause of variable and leads to non-reliable test results. A secondary cause of HPC result variability is likely to be low levels of instrument sensitivity, as most systems currently on the market were designed to measure the permeability properties of normal concrete [11,12]. As HPC permeability levels are typically low, relative performance differences become difficult to detect.

Against this background, the aim of this study is to propose a suitable pre-conditioning regime for air permeability tests and to assess the ability of the Autoclam test to identify relative HPC performance levels. In order to achieve this aim, concrete specimens were dried and conditioned before moisture conditions were examined using relative humidity and electrical resistance measurements. Air permeability was measured using the Autoclam test method at different moisture conditions. In addition, standard water penetration laboratory-based testing was carried out to examine relative HPC permeability and to enable comparisons with Autoclam air testing results.

2. Experiment programme

2.1. Materials and concrete mixes

Based on previous experimental work undertaken at Queen's University [8,13], two HPCs and one normal concrete were considered as part of this study. The mix compositions are reported in Table 1.

With a water/binder ratio of 0.68, the reference, normal concrete contained Portland cement as a binder material only. Similarly, the first HPC considered (labelled HPC1-PC) contained Portland cement only but at a water/binder ratio of 0.30. Typical for HPCs [1], the second HPC considered contained a ternary binary

Table 1
Concrete mix proportions.

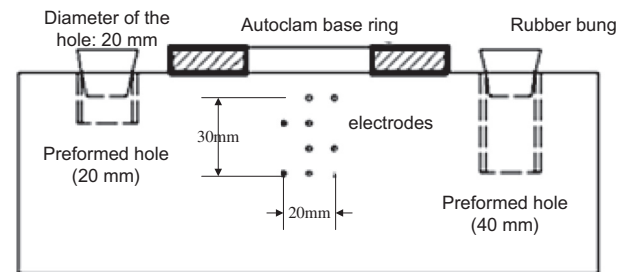
Constituents	Concrete mix		
	Normal concrete (NC)	HPC1 (PC)	HPC2 (MF)
PC ^a (kg/m ³)	375	485	352
Microsilica (kg/m ³)	0	0	36
PFA (kg/m ³)	0	0	97
Sand (kg/m ³)	625	689	652
Coarse aggregate (kg/m ³)	1136	1150	1150
Water (kg/m ³)	256	145	145
Superplasticiser ^b (%)	0	1.3	1.5
Water/binder ratio	0.68	0.30	0.25

^a PC was CEM II contained 85 ± 0.5% Portland cement clinker and 15 ± 0.5% limestone powder.

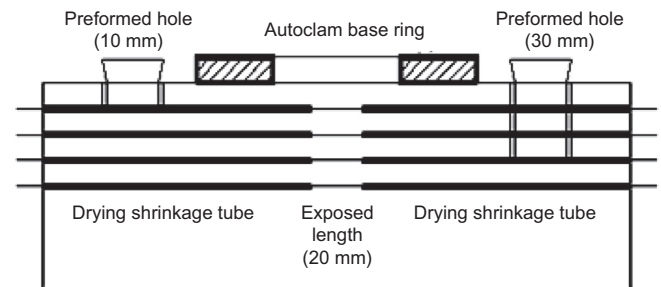
^b Polycarboxylic acid based polymer superplasticiser, as percentage of binder content.

Table 2
General properties of concrete in this work.

Property	Concrete mix		
	NC	HPC1-PC	HPC2-MF
Air content (% by volume)	0.7	1.3	0.8
Slump (mm)	180	205	235
28 day compressive strength (MPa)	24.1	78.6	80.2
56 day compressive strength (MPa)	33.7	86.3	85.3



(a) The sectional view of the specimen



(b) The sectional view of the specimen

Fig. 1. Details of the test specimens.

blend of Portland cement, micro silica (MS) and pulverised fuel ash (PFA). The water/binder ratio in this case was 0.3. In all cases the cement used was type CEM-II conforming to BS EN 197: 2000 [14]. The PFA, conforming to BS EN 450: 2005 [15], was sourced from Kilroot Power station in Northern Ireland and the MS in slurry form, conforming to BS-EN 13263-1: 2009 [16], from Elkem. A polycarboxylic acid-based polymer superplasticiser, commercially available as Chemcrete HP3, was additionally used for the HPC mixes to maintain a constant slump range.

The fine aggregate was medium graded natural sand and the coarse aggregate both 10 and 20 mm crushed basalt used in equal proportions. The moisture condition of the aggregates was controlled by pre-drying in an oven at 105 ± 5 °C for 24 h followed by cooling to 20 ± 1 °C for one day before mixing.

Each concrete mix was tested for air content [17], slump [18] and compressive strength [19], the results of which are shown in Table 2. The disparity between the normal concrete and HPC is evidenced by the variation in 28-day compressive strength, which was approximately 24 N/mm² for mix NC and 80 N/mm² for both HPC mixes.

2.2. Specimen preparation and testing

For each concrete mix, fifteen 230 × 230 × 100 mm slabs were manufactured for both moisture-conditioning and air permeability testing, and 100 mm cube specimens for compressive strength testing. The slab specimens contained pre-formed cavities at depths of 10, 20, 30 and 40 mm and embedded four pairs of stainless steel electrodes for testing relative humidity and electrical resistance (see Fig. 1). The cavities were sealed by rubber plugs to ensure that the reading in the hole is not affected by the ambient conditions.

Concrete mixing was undertaken in accordance with BS 1881, Part 125: 2005 [20] and the fresh concrete was assessed for consistence (slump test) and air content. Concrete was compacted in moulds in two layers using a vibrating table, covered with wet hessian and placed in a constant temperature room (18 ± 2 °C).

Table 3

Slab specimen curing, preparation, drying/conditioning and testing regime.

Curing regime/specimen preparation				Comments
Sample curing and preparation	<i>Specimens de-moulded</i>			
	3 days	20 ± 1 °C	Constant temperature water bath	Initial curing period
	90 days	20 ± 1 °C	Wrapped in polythene sheet and located in a constant temperature room	Intended to remove influence of hydration on subsequent test results
	3 days	Sides painted with epoxy paint and specimens saturated by incremental immersion		Sample preparation to prohibit future moisture transport
<i>Testing: Electrical resistance</i>				
Drying	1, 2, 3, 4 or 5 weeks	50 ± 1 °C RH 35%	Drying cabinet.	Initial moisture profiling. Assessment of drying time on performance
	1 day	~20 °C	Wrapped in polythene and cooled to room temperature.	Pre-testing conditioning
<i>Testing: Relative humidity, electrical resistance and air permeability</i>				
Conditioning	2 weeks	50 ± 1 °C	Wrapped in polythene sheet placed in drying cabinet	Main testing programme Undertaken to achieve uniform moisture distribution
	1 day	~20 °C	Wrapped in polythene and cooled to room temperature	Pre-testing conditioning
<i>Testing: Relative humidity, electrical resistance and air permeability</i>				
Sample preparation	3 days	100 mm diameter cores extracted		Main testing programme Sample preparation
Sides painted with epoxy paint and specimens saturated by incremental immersion.				
Testing: Water permeability				Confirmation testing

All specimens were de-moulded after 24 h and placed in a constant temperature water bath (20 ± 1 °C). Cube specimens were removed after 28 and 56 days and tested for compressive strength. Slab specimens were removed after three days, wrapped in polythene sheets and relocated to a constant temperature room (20 ± 1 °C) for 90 days to remove any influence of hydration on subsequent test results. After this 90 day period, slab sides were painted with three coats of an epoxy paint to prohibit moisture transport. Following recommended procedures the slabs were then saturated by incremental immersion [13,21], involving exposure of half their thickness to water to allow both water absorption and air removal. After 24 h a further quarter of the specimen depth was exposed to water before complete immersion after 48 h.

At the end of the saturation period, initial electrical resistance measurements were taken and slabs were placed in a drying cabinet at a temperature of 50 ± 1 °C and relative humidity of 35%. Five different drying periods of 7, 14, 21, 28 and 35 days were considered, after which three slabs for each mix were removed from the oven, wrapped in polythene sheet and cooled to room temperature (around 20 °C) for 1 day. Air permeability, relative humidity and electrical resistance measurements were then taken. The slabs were then conditioned by being wrapped in polythene sheet and place back into the drying cabinet (50 ± 1 °C) to encourage uniform internal moisture distribution. By the end of 2 weeks of this conditioning regime, the sealed specimens were transferred to a testing laboratory (20 ± 1 °C) and re-tested for air permeability, relative humidity and resistance.

After carrying out the last set of Autoclave air permeability tests, three 100 mm diameter cores were cut from the specimens and side surfaces coated with epoxy resin to prevent any lateral flow of air during testing. The samples were then saturated by incremental immersion as described above and then tested for water penetration in accordance with BS EN 12390-8: 2000 [22]. The curing, preparation, drying/conditioning and testing regime for the slab specimens is summarised in Table 3.

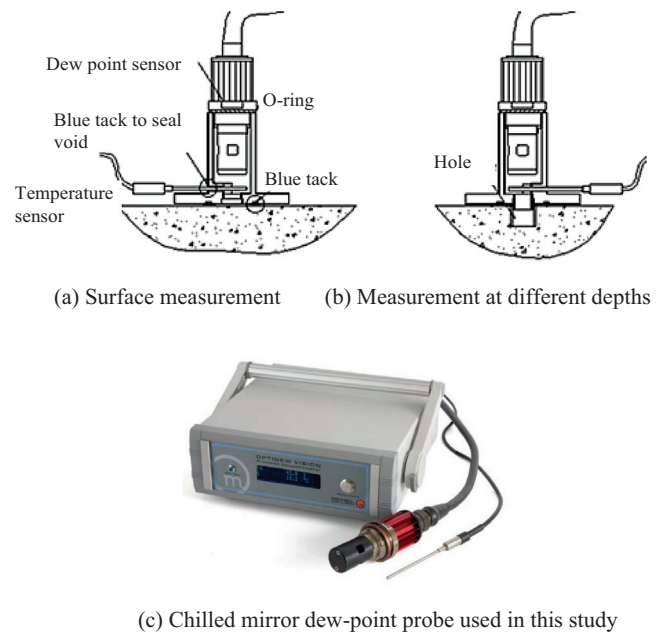
2.3. Test methods

2.3.1. Relative humidity (RH)

A commercially available, chilled mirror dew-point probe was used to measure RH. The test arrangement for RH measurements followed procedures developed previously by Basheer and Nolan [23] and involved testing at the surface and at depths of 10, 20, 30 and 40 mm within preformed cavities (see Fig. 2). Following recommendations, stable results were obtained after one hour. Each RH value plotted in Figs. 6 and 7 is the average of two separate measurements.

2.3.2. Electrical resistance

As variation of electrical resistance is capable of representing the movement and distribution of moisture in concrete [24], measurements between electrodes embedded at 10, 20, 30 and 40 mm below the surface (see Fig. 1) were recorded using a TINSLEY resistance meter (Fig. 3). The average of two measurements was reported.

**Fig. 2.** Method of measuring RH at surface and in the preformed cavities.

2.3.3. Autoclave air permeability

The Autoclave permeability system was developed by Queen's University Belfast to measure the sorptivity, air permeability and water permeability of concrete cover on site [12,25]. In this study a standard base ring with an internal diameter of 50 mm was used to isolate a test area on the surface of the concrete blocks, before the instrument was pressurized manually using a syringe attached to the base of the testing system (see Fig. 4). At the desired pressure of 0.5 bar, the test commenced automatically and pressure decrease monitored every minute for 15 min. The natural logarithm of air pressure was plotted against time, with the slope of the last five data points reported as the air permeability index (API) in ln(bar)/min.

2.3.4. BS-EN water penetration

Water permeability coefficients were determined using the water penetration test according to BS EN: 12390-8: 2000 [22] (see Fig. 5). Once the test arrangement was pressurized with air to 8.5–9 bar, the hydraulic cylinder was opened and a constant test pressure of 7.3 bar was applied for three days. At the end of the test,

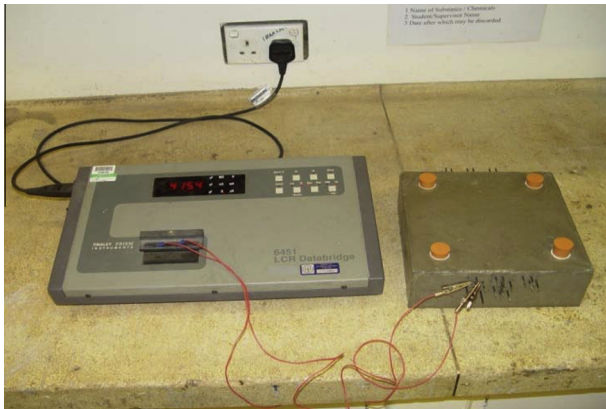


Fig. 3. Electrical resistance measurement using a resistance meter.



Fig. 4. The autoclave permeability system and syringe for applying air pressure.

specimens were split open and the depth of water penetration measured. Three 100 mm diameter cores were extracted from the slab specimens and tested for each concrete mix.

3. Results and discussion

3.1. RH after drying and conditioning

RH results at different depths from the exposed surface after drying for periods of 1–5 weeks are shown in Fig. 6. As anticipated, RH values generally decreased with increasing periods of drying [21]. It was also found that near-surface RH values were lower in comparison to interior results, meaning that drying conditions caused the development of moisture gradients. Fig. 6 also

highlights that the two HPCs considered had lower (on average 42%) RH values than the normal control concrete (around 60%) after 1 week of drying. This difference is most likely reflecting the fact that a fully saturated state is frequently difficult to reach for HPC [26,27], despite having used the incremental immersion method. As the control concrete inherently comprised relatively larger pores, it is likely that its moisture content was higher at the start of drying, thereby explaining its higher RH values.

Fig. 6 additionally reveals that between 2 and 5 weeks of drying, the reduction of RH was around 5% slower for the HPC relative to the control. This can be explained by the combined effects of water vapour transport under different RH ranges and the different characteristics of the pore structures. For HPC, finer and less connected pore structures make moisture transport slower [1]. Furthermore, most of the HPC's RH readings were below 70%, indicating that most of the coarse capillaries were empty [26]. Research shows that further drying might have caused the removal of adsorbed moisture and some interlayer moisture when the RH was lower than 65% [28]. The behaviour of adsorbed moisture is quite different from free capillary moisture due to its high viscosity, with more energy required for its removal from the solid surface; a process which is very slow. As a result, drying rates of the HPC were lower at low RH values.

In agreement with previous research [13,29,30], Fig. 6 also highlighted that RH gradients existed even after 5 weeks of drying, suggesting that the drying regime used was not sufficient to establish equilibrium moisture conditions. In order to re-distribute moisture in this study, slabs were subsequently wrapped in a polythene sheeting and relocated into an oven at $50 \pm 1^\circ\text{C}$ for 14 days. The resulting relationships between RH at different depths from the exposed surface are re-plotted in Fig. 7, which shows that this approach effectively removed RH gradients almost entirely. It was also noted that, after 4 weeks of drying, there was no significant difference in RH for the normal concrete, but the moisture distribution was slightly different with each drying period for the HPC. As air permeability is not expected to vary when the RH of the concrete is between 40% and 60% [31], it was subsequently decided that the drying and conditioning procedures proposed were applicable to HPC prior to undertaking air permeability measurements.

3.2. Electrical resistance after drying and conditioning

Electrical resistance ratios (R_t/R_{i40}) for each depth after drying are plotted in Fig. 8. R_t/R_{i40} is regarded as the relative change of resistance, with R_{i40} the resistance value at 40 mm from the exposed surface after immersion for 72 h and R_t the resistance value at each recording depth after different drying periods.

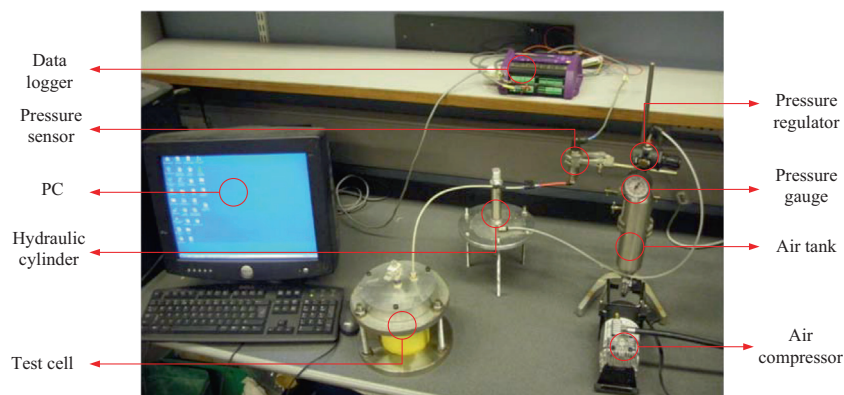


Fig. 5. Water penetration test set-up to BS EN: 12390-8.

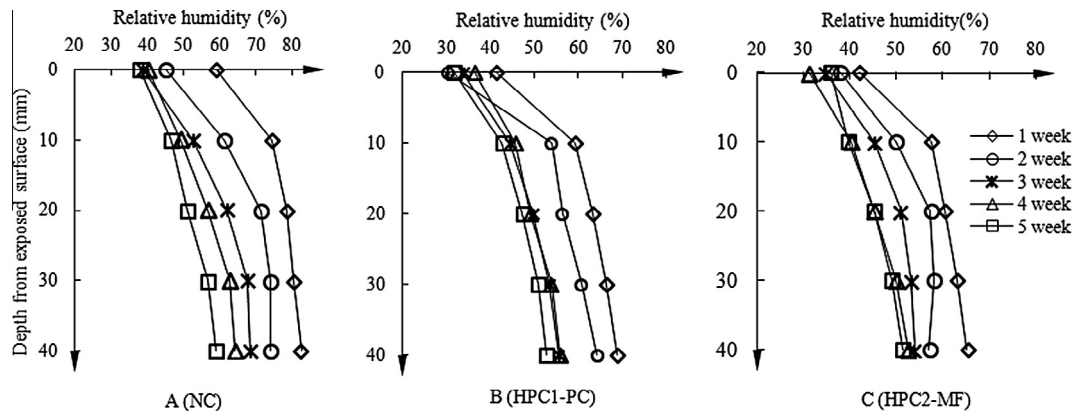


Fig. 6. Relative humidity results at different depths after drying ($50 \pm 1^\circ\text{C}$, RH 35%) for different periods (1–5 weeks).

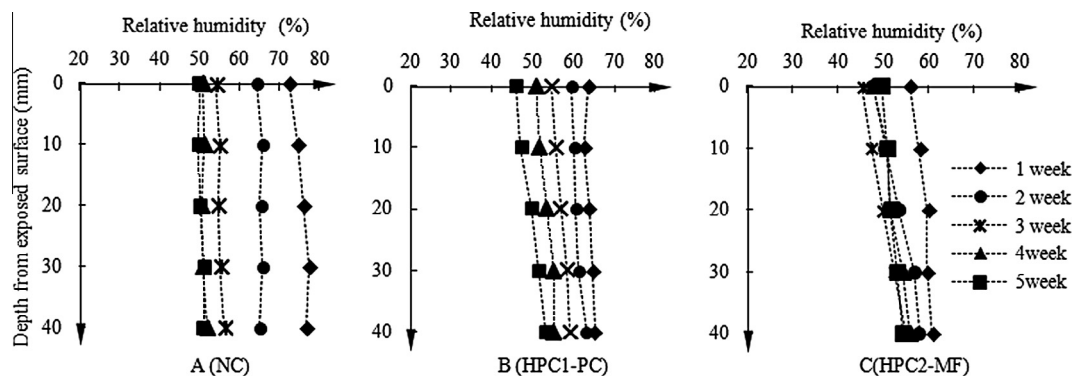


Fig. 7. Relative humidity at different depths after drying ($50 \pm 1^\circ\text{C}$, RH 35%) for different periods (1–5 weeks) and conditioning (wrapped in polythene, 50°C) for 2 weeks.

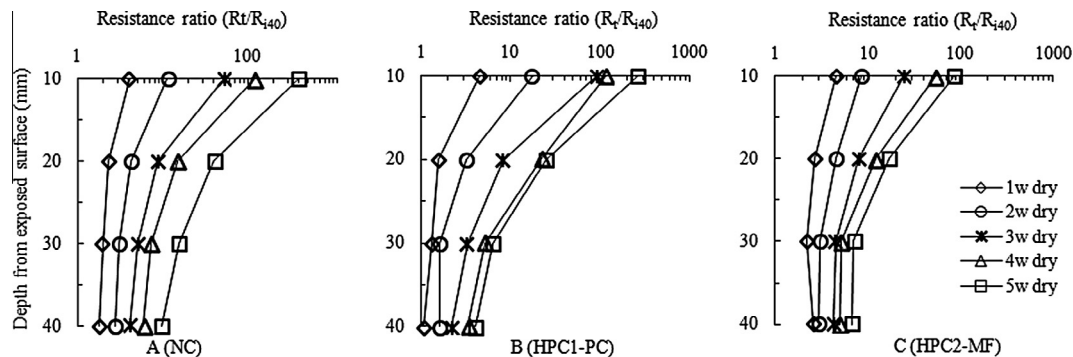


Fig. 8. Electrical resistance ratio variations at different depths after drying ($50 \pm 1^\circ\text{C}$, RH 35%) for different periods (1–5 weeks).

As expected, electrical resistance ratios generally increased with increasing drying periods, with more marked changes noted closer to specimen surfaces. This trend is attributable to the increased loss of moisture in this region as evidenced by the RH values plotted in Fig. 6.

Furthermore, minor increases in resistance ratio were noted during the first two weeks of drying, followed by more significant increases thereafter. As such, it can be inferred that prolonged drying causes a loss of moisture connectivity within capillary pores. This trend is the converse of that noted for the RH results plotted in Fig. 6. It has already been explained that during the drying process only free capillary water was lost initially, whilst adsorbed water was removed at the later stages of drying. When adsorbed moisture is lost, electrical current has to pass through the solid

phases of hydration, which have much higher resistivity. Therefore, the removal of adsorbed moisture could be considered as the reason for the sharp increases noted in electrical resistance [21,32].

Fig. 9 displays the variation of resistance ratio after both drying and conditioning specimens. Clearly, while conditioning decreased resistance ratio gradients, they could not be removed entirely. This behaviour shows an obvious contrast with Fig. 6. It is believed due to the combined effects of moisture redistribution and further hydration on resistance, whereas RH measurements only represent moisture distribution and is not significantly affected by further hydration. The effective redistribution of moisture during the pre-conditioning stage is illustrated in Fig. 10, which also shows moisture distributions after drying and conditioning. It is known that as hydration progresses, electrical resistance ratios generally increase

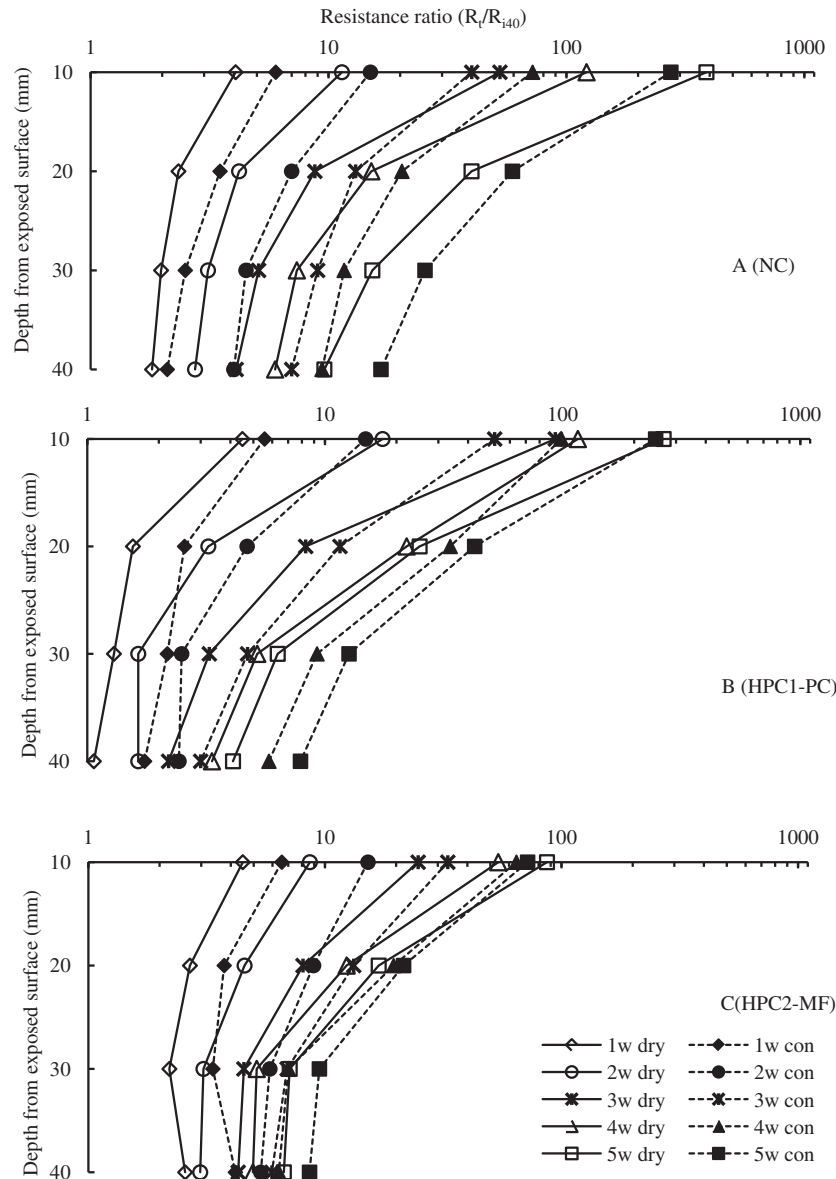


Fig. 9. Comparison of variation of resistance ratio after drying ($50 \pm 1^\circ\text{C}$, RH 35%) for different periods (1–5 weeks) and conditioning (wrapped in polythene, 50°C) for 2 weeks.

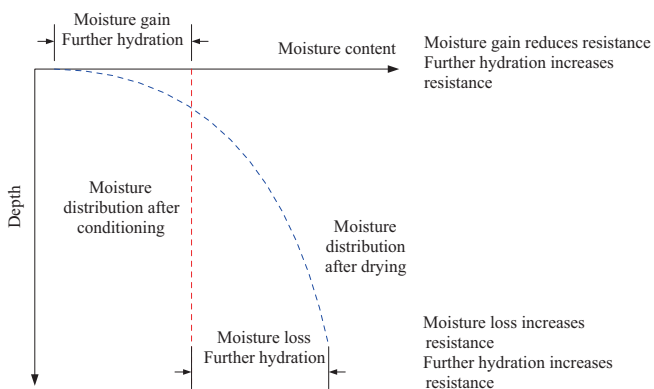


Fig. 10. Illustration of moisture distribution after drying and conditioning.

[30], whereas the internal RH depends on the extent of redistribution of moisture inside the concrete relative to the surface. During drying, internal moisture moves towards the surface where there

are significant moisture losses due to evaporation with associated reductions in the degree of hydration. The combined effect is to increase the resistance ratio at the near surface, which increases with depth from the surface as depicted in Fig. 8. However, two opposing effects exist during the conditioning process in the near surface region; namely moisture gain and further hydration. During redistribution, free moisture increases near the surface due to its transport from inner regions to the near surface region, but potentially with no further improvements in the degree of hydration at this stage. As a result, the resistance ratio near the surface shows a combined reduction. On the other hand, resistance ratios at deeper levels after conditioning show greater values (as in Fig. 10) as further hydration occurs at these depths, although there would be some loss of moisture during redistribution.

3.3. Influence of drying on reliability of Autoclave air permeability tests

Autoclave air permeability testing was used to investigate effects of moisture gradient and its suitability for ranking HPC performance was assessed. Fig. 11, which plots the effect of drying

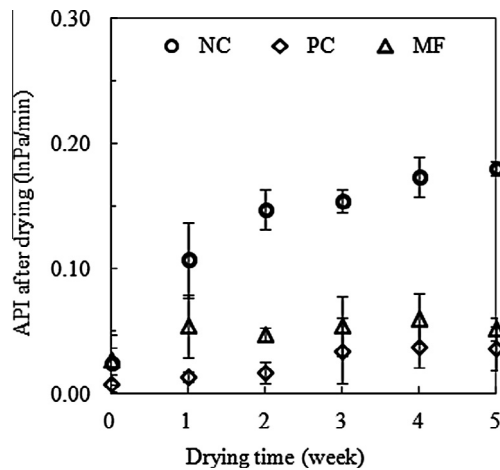


Fig. 11. Effect of drying on air permeability index (API).

time on API, shows no obvious differences between normal concrete and HPC prior to oven drying (i.e. at drying time of zero weeks). This implies that the effect of moisture content on air permeability dominates over effect of concrete type. As such, and in line with previous research [13,21,22], this study confirms the inappropriateness of classifying concrete air permeability when the effect of moisture content has not been addressed.

Fig. 11 also illustrates a sharp rise in API for normal concrete after 2 weeks of drying, with comparatively small increases thereafter. For normal concrete, 2 weeks of drying resulted in RH values around 70% at 40 mm (the effective test depth for Autoclam). This means that most pores were free from capillary water in this region and hence the effect of moisture on API has been eliminated [12,32,33]. In the case of HPC, however, there were no significant changes in API beyond 1 week of drying. Indeed, API values for HPC had almost no variation before 3 weeks of drying and only minor increases thereafter. Correspondingly, all of the RH values of HPC above 40 mm (see Fig. 6) were around 50%. This implies that the influence of moisture on API is relatively small [22,31].

The relationship between API after specimen drying and conditioning is shown in Fig. 12, which clearly illustrates lower values after conditioning. Both moisture redistribution and further hydration are considered to be the reasons for this slight decrease in API after conditioning [13]. However, the influence is small for concretes dried beyond 3 weeks, illustrating that any effect of moisture redistribution on API was found to rely on the amount of

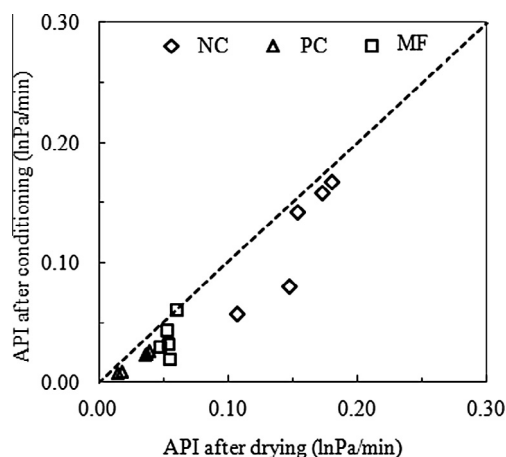


Fig. 12. Relationship of air permeability index (API) after drying (50 ± 1 °C, RH 35%) for different periods (1–5 weeks) and conditioning (wrapped in polythene, 50 °C) for 2 weeks.

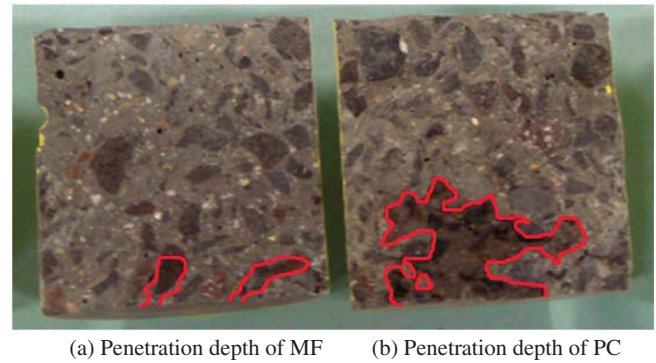


Fig. 13. Typical water penetration results to BS EN: 12390-8.

free moisture available in specimens. For normal concrete with relatively high free moisture contents, redistribution can reduce API significantly due to the increase in near-surface moisture content. This effect was found to decrease when concretes were dried for 2 weeks. In the case of HPC, it was found that the reduction of API was insignificant at all drying conditions.

It is evident that API values for the two HPCs were very similar, even after a drying period of 5 weeks which has effectively removed any influence of moisture. Possible explanations for this phenomenon are that, firstly, no permeability differences existed between the two HPCs and, secondly, that the test method was not sufficiently sensitivity to distinguish any potential differences.

The standard BS-EN laboratory water penetration test was performed to clarify this point as shown in Fig. 13. From this Fig. it can be clearly seen that the penetration depth of HPC mix MF (average 13.6 mm) was appreciably lower than that of mix PC (average 35.7 mm), suggesting that significant permeability differences did exist between the two mixes.

From these findings it may be concluded that while the conventional Autoclam air permeability test is suitable for assessing normal concrete performance, it is not sufficiently sensitive to detect relative HPC performance levels. This finding is perhaps not surprising given that the test method was developed primarily for testing normal concrete [12,33,34].

3.4. General discussion

It is generally accepted [35–37] that concrete drying regimes have the potential to damage the material's microstructure. Numerous researches have reported, for example, that pore structures will not maintain stability under high temperature [5,12,38,39]. As such, this aspect was examined in this study using analytical methods and through comparisons with relevant information in the literature. Potential material damage was checked by studying the behaviour of moisture transport under different RH ranges.

As RH reaches 65%, most capillaries contain no free moisture and adsorbed moisture loss commences, albeit with minimal effect on gel structure. Once RH is below 40%, however, interlayer moisture loss will occur [40], resulting in the collapse of fine pores and the formation of coarse pores. This can generate micro-cracking in the paste matrix and lead to more connected flow paths [12,36]. The influence of RH on air permeability has been previously examined by Parrott [31], who plotted permeability as a function of RH, relative to permeability at 60% RH (K_{RH}/K_{60}). Three performance regions were identified from this work as follows:

- (1) At RH between 100% and 60%, the influence of moisture on permeability is significant and examinations of air permeability in this range should be avoided.

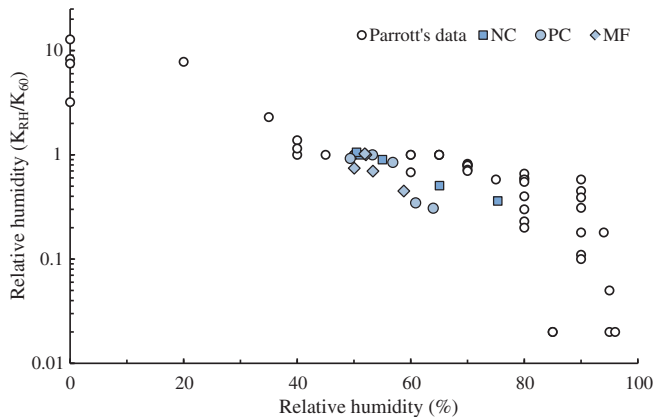


Fig. 14. Effect of relative humidity on permeation data.

- (2) At RH between 60% and 40%, air permeability measurements are generally constant, reflecting the fact that moisture effects have been removed.
- (3) At RH between 40% and 0%, drying becomes aggressive and the microstructure of the paste matrix is significantly changed, particularly below RH 20%. As such, pre-conditioning regimes should avoid drying concrete below this point.

The results from the current study are compared to Parrott's data as shown in Fig. 14, which illustrates very similar trends. As all RH values were maintained above 40% in the study reported in this paper, it may be concluded that the moisture condition in the gels was not significantly affected by the drying regime and, as such, no significant damage occurred in the gel phases.

4. Conclusions

Based on analysis of data obtained, the following conclusions have been drawn:

- (i) After 28 days of drying, no significant difference in RH values was observed between the three different concretes tested, albeit that initial rates of moisture loss differed. Results further suggest that moisture gradients are effectively removed for all mixes after sealed conditioning for 2 weeks.
- (ii) Gradients of electrical resistance after moisture redistribution persist, which is not necessarily due to moisture distributions, but most likely caused by porosity gradients and variations in degree of hydration.
- (iii) While the two HPCs examined exhibited different permeability based on the results of the BS-EN water penetration test, the conventional Autoclam air permeability test with a 50 mm diameter base ring was not able to distinguish these differences. This suggests that the sensitivity of this method merits improvement at the performance levels typical for HPC.
- (iv) It was concluded that for normal concrete, 14 days of drying was sufficient to negate effects of moisture gradients on API, due to removal of free moisture near the concrete surface. For HPC, the values of API are unable to clarify the influence of moisture due to the non-sufficient sensitivity of the conventional Autoclam, but the values of RH indicate that 3 weeks of drying are enough to remove capillary moisture within HPC.

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